

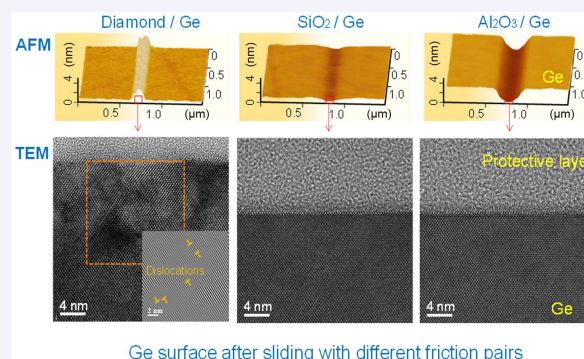
Defect-free removal of monocrystalline germanium

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ABSTRACT: Germanium (Ge) serves as an emerging alternative for smaller devices with dense features and is of significance in the microelectronics industry. Since surface defects can degrade directly the performance of Ge-based devices, it is essential to reduce or eliminate machining damage on Ge substrate. In this study, the defect-free material removal behaviors of Ge were investigated by a series of nanoprocessing with SiO₂ and Al₂O₃ tips using atomic force microscope (AFM), and the tests with a diamond tip were performed for comparison. Material removal on Ge surface exhibited the dependence on ambient humidity whatever SiO₂, Al₂O₃ or diamond tip was employed, while the sliding with Al₂O₃ tip can lead to the largest quantity of surface removal. Combined with micro-Raman spectra and high-resolution transmission electron microscopy (HRTEM) detection of the worn area, it was confirmed that no defects existed on the Ge surface during the material removal process by SiO₂ and Al₂O₃ tips. It also demonstrates that defect-free material removal occurred on the Ge surface under contact pressures far below the yield strength of Ge, which can be attributed to water-participated mechanochemical reactions. Further analysis showed that the presence of hydroxyl ions and radicals weakening Ge-Ge backbone bonds could dominate the defect-free removal behavior by SiO₂ or Al₂O₃ tip sliding. This study may shed light on the mechanochemical removal mechanism and provide the guidance for defect-free processing of Ge.

KEYWORDS: monocrystalline germanium (Ge); defect-free material removal; microstructures; atomic force microscope (AFM); mechanochemical removal



Ge surface after sliding with different friction pairs

1 Introduction

Germanium (Ge) has aroused particular concern in technical fields such as quantum processors, solar cells, and photoelectric detection because of its unique physical and photoelectric conversion efficiency [1–4]. Given its superior carrier mobility and narrow band gap, Ge has transitioned from an emerging alternative to an indispensable channel material, driving performance enhancements in highly scaled devices down to sub-5 nm and 3 nm technology nodes [5, 6]. Accordingly, surface and subsurface damage of monocrystalline Ge wafers can degrade the performance of subsequent functional devices, and it is necessary to reduce the occurrence of defects in surface manufacturing [7]. Many factors can contribute to mechanochemical removal process during chemical mechanical polishing (CMP) and further influence the quality of surface finish, such as abrasive properties (e.g., mechanical properties, surface chemistry, etc.), loading parameters (e.g., normal load/contact pressure, speed, etc.), and environmental conditions (e.g., relative humidity (RH), vapor, etc.) [8, 9]. Atomic force microscope (AFM)-based single asperity contact tests were conducted to simulate complex interfacial contact state, tribochemical reaction and abrasive removal with

the advantage of *in situ* and quantitative investigations [10–12], which can be used to quantify the defects and removal efficiency resulting from polishing process. The depth of subsurface damage on Ge surface was found to be shallower at a lower temperature after being polished by diamond lapping pad at various ambient temperatures, but the defects still existed [13]. Numerous studies have focused on using silica along with different additives to reduce defects on Ge surface [14–16]. Generally, H₂O₂ was applied as the oxidizer for etching, which has also been proven to be the most effective addition for material removal [17]. Nevertheless, the roughness on Ge surface was found to increase with H₂O₂ and colloidal silica concentration but decreased with pH [18]. It was shown that Ge exhibited significant dissolution in alkaline pH conditions but not in acidic and neutral pH conditions, which could be attributed to the existence of powerful oxidizing hydroxyl radicals [13]. Besides, Gupta et al. [19] found that the formation of Ti–O–Ge bonds due to the structural similarity of polishing surfaces and abrasives resulted in a higher removal rate of Ge when using rutile abrasives.

In addition to the reports on Ge CMP using silica-based slurry, whether other particles can bring a better polishing effect is still unclear [16]. The mechanochemical interaction is mainly ascribed

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to the formation of chemical bonding bridge between the substrate and abrasive particles, which is assisted by the presence of water meniscus on interface [20, 21]. Relative humidity and sliding velocity were demonstrated to affect dynamic formation of bond bridges and rupture rate of interface bonds during relative sliding of friction pairs. The mechanochemical interactions strongly depend on chemical activity of two materials interacting at the tribological interface. Meanwhile, some abrasive particles (silica (SiO_2), alumina (Al_2O_3), etc. [22]) can participate as reactants in the material removal of tribological interfaces [17, 19]. For instance, the sliding of SiO_2 or Al_2O_3 tip can bring about mechanochemical removal of GaAs surface, and defect-free matrix can be observed on the bottom of the scratch [23]. As a result, the removal on the Ge surface under the action of different abrasive particles even remains full of some mystery, and corresponding investigation is of great significance for understanding the interface tribochemical mechanism and improving the polishing of Ge surface. Particularly, in practical CMP and related micro/nano-manufacturing processes, it is widely recognized that environmental humidity profoundly alters interfacial contact mechanics and triggers localized water-assisted tribochemical reactions [20]. These confined water molecules do not merely act as physical lubricants; rather, they actively participate in interfacial mechanochemical behaviors. Consequently, variations in environmental moisture can fundamentally alter both the material removal efficiency and the evolution of subsurface damage. Therefore, a systematic nanoscale investigation into the humidity-dependent mechanochemical interactions is imperative for elucidating the damage-free planarization mechanism of monocrystalline Ge.

In this study, defect-free removal of monocrystalline Ge was achieved by SiO_2 and Al_2O_3 tip scratching on an AFM with an environmentally controlled system. For comparison, the scratches were produced by a diamond tip. The relationship between the chemical activity of the friction pair and defect-free removal behaviors of Ge surface was investigated. Furthermore, combined with the detections by micro-Raman spectroscopy and high-resolution transmission electron microscopy (HRTEM), the nanoscale material removal by SiO_2 and Al_2O_3 tips was found to be dominated by mechanochemical behavior at the contact interface.

2 Materials and methods

N-type Ge (100) wafers (Hefei Kejing Materials Technology Co., Ltd., China) were used for tip processing. Before the tests, Ge samples were first immersed in hydrochloric acid (~10 wt% HCl in aqueous solution) and etched for at least 2 min to remove the oxide layer and surface contaminants [24]. Then all Ge samples were ultrasonicated (the ultrasonic frequency is about 40 kHz) in acetone, ethanol, and deionized (DI) water in sequence to avoid the effect of residual etchant on subsequent tests. All nanoprocessing was performed under line- and area-scratch modes by different tips using an atomic force microscope (E-

Sweep, Hitachi Instruments Inc., Japan) equipped with an environmentally controlled system. SiO_2 and Al_2O_3 tips with curvature radii (R) of $\sim 1\ \mu\text{m}$ and spring constant (k) of 16 N/m were employed for the tests, respectively. In addition, a diamond tip with $R \approx 0.5\ \mu\text{m}$ and $k \approx 200\ \text{N/m}$ was used for comparison. The scratches were produced after 500 line-scratch cycles (N) at a scanning speed (v) of $4\ \mu\text{m/s}$ under a normal load (F_n) of $4\ \mu\text{N}$ with a relative sliding amplitude (A) of $2\ \mu\text{m}$. All topographic images of tip-processed areas were obtained in vacuum ($< 3 \times 10^{-4}\ \text{Pa}$) by a $R \approx 20\ \text{nm}$ silicon nitride tip (MSCT, Bruker, USA).

To further understand the potential phase transformation on the Ge surface during the nanoprocessing tests, $20\ \mu\text{m} \times 20\ \mu\text{m}$ scratches generated by diamond, SiO_2 , and Al_2O_3 tips, were characterized by laser confocal micro-Raman spectroscopy (LabRAM HR Evolution, Horiba, Japan) with an incident beam of 632.8 nm focused to a spot size of $\sim 1\ \mu\text{m}$. Moreover, to further explore microscopic defects and reveal the mechanism of the material removal by different tips on the Ge surface in humid air, the cross sections of the removal tracks were examined by high-resolution transmission electron microscope (HRTEM; Tecnai G2 F20, FEI, USA).

3 Results and discussion

3.1 Nanoprocessing on the Ge surface in humid air

The material removal process on monocrystalline Ge surface is often accompanied by serious defects [25]. Therefore, surface finishing is usually conducted under light contact pressure to reduce the generation of defects. In this study, the nanoprocessing tests were applied on the Ge surface by three friction pairs (diamond/Ge, SiO_2 /Ge, and Al_2O_3 /Ge) under a normal load of $4\ \mu\text{N}$. The corresponding contact pressure according to Hertz's theory calculation, about 3.1 GPa for the friction pair of diamond/Ge, 1.6 GPa for SiO_2 /Ge, and 1.8 GPa for Al_2O_3 /Ge, has not reached the yield strength of Ge, which is 3.5 GPa for monocrystalline Ge [26]. For comparison, the removal process with different tips was conducted under the same contact pressure as above. Nevertheless, when the same contact pressure as SiO_2 /Ge or Al_2O_3 /Ge was applied to the friction pair of diamond/Ge, no significant material removal or hillock formation was observed on the Ge surface.

AFM topographic images displayed removal tracks from three friction pairs on the Ge surface in air with a relative humidity (RH) of $\sim 40\%$, as shown in Fig. 1. It can be observed that SiO_2 and Al_2O_3 tips caused material removal from the Ge surface. Such material removal on the Ge surface under purely elastic contact conditions can be ascribed to the synergistic effect of mechanical and chemical reactions. Specifically, driven by the applied shear stress, the hydroxyl groups ($-\text{OH}$) on both the tip and the Ge surface dynamically interact to form interfacial bridging bonds (e.g., $\text{Ge}-\text{O}-\text{Si}$ or $\text{Ge}-\text{O}-\text{Al}$). As the tip continuously slides, the rupture of these bridging bonds facilitates the atomic-scale extraction of Ge from the substrate, culminating in the formation

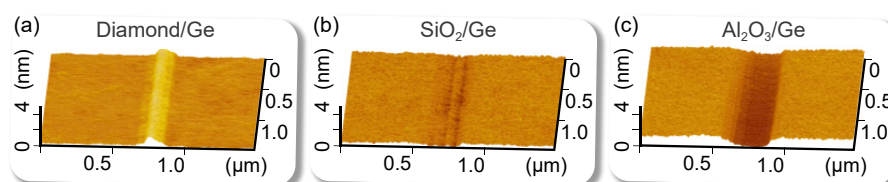


Fig. 1 AFM topographic images of the tip-processed Ge surfaces. The protrusive hillock (a) was created by a diamond tip, while the grooves (b, c) were produced by SiO_2 and Al_2O_3 tips, respectively.

of distinct removal grooves [27]. This mechanochemical mechanism is similar to the surface defect-free removal process of silicon belonging to the same group [28]. Counterintuitively, friction-induced hillocks were generated in the diamond tip scratched area under a light normal load. The formation of hillocks was mainly attributed to amorphization induced by mechanical shear [29, 30]. Given that material removal occurred with SiO₂ and Al₂O₃ tip processing instead of diamond tip under purely elastic contact conditions, it can be inferred that mechanochemistry may play a critical role in material removal on monocrystalline Ge surface.

3.2 Material removal on the Ge surface in different humidities and DI water

To further study the material removal behavior on monocrystalline Ge surface by diamond, SiO₂, and Al₂O₃ tips, the tests were performed in a variety of humidity air (~40%, 60%, and 80% RH) and DI water. For comparison, Fig. 2 shows AFM images of scratches under the same parameters by different tips. It can be observed that the hillock was gradually removed with increased humidity, which appears to be a mechanical removal behavior. The removal on the Ge surface against SiO₂ tip sliding increased significantly with the humidity, while most of the removal was in DI water. Nevertheless, the material removal on the Ge surface by Al₂O₃ tip sliding was much greater than that by SiO₂ and diamond tip. While in DI water, all three tips can cause intense removal on the Ge surface.

In Fig. 3(a), the corresponding cross-sectional profiles of the scratches on the Ge surface further demonstrate the material removal change with the humidity. It can be noticed that the height of the scratch produced by diamond tip decreased from an initial value of 1.2 nm (hillock) to a final value of -0.3 nm (groove) with the humidity, while it decreased to -1.7 nm in DI water. Besides, the removal depths generated by SiO₂ tip in humid air were all ~0.3 nm. By contrast, the scratches formed by Al₂O₃

tip changed slightly, and their depths increased from 1.1 to 1.5 nm. While in DI water, the removal depth increased to 2 nm by SiO₂ tip and 1.9 nm by Al₂O₃ tip, respectively. The results show that the material removal of Ge by SiO₂ and Al₂O₃ tips may undergo different removal paths compared to that by diamond tip in humid air. It is deduced that SiO₂ and Al₂O₃ tips may bring different tribochemical effects to the Ge surface.

To reveal the relationship between the material removal and the three friction pairs, the changes in removal depth (Fig. 3(b)) and removal volume (Fig. 3(c)) of scratches on the Ge surface were plotted for further comparison. It can be seen from Fig. 3 that the Al₂O₃/Ge pair experienced the severest material removal with the deepest and the largest removal track in humid air, followed by SiO₂/Ge. As the humidity increases, such different quantities of material removal can be ascribed to different mechanochemical mechanisms depending on friction pairs. For the friction pair of diamond/Ge, the protuberance volume gradually decreased sequentially from $0.4 \times 10^6 \text{ nm}^3$ (hillock), $0.13 \times 10^6 \text{ nm}^3$ to $-0.02 \times 10^6 \text{ nm}^3$ (groove) with increased humidity, and finally reached $-0.3 \times 10^6 \text{ nm}^3$ in DI water. The hillock formation may stem from mechanical shear-induced phase transformation [29], and the decrease in hillock height with humidity may be partly attributed to the removal of water-soluble oxides on the hillock surface during the sliding process [31, 32]. The removal volume on the Ge surface by Al₂O₃/Ge pair increased slightly from $0.64 \times 10^6 \text{ nm}^3$, $0.7 \times 10^6 \text{ nm}^3$ to $0.75 \times 10^6 \text{ nm}^3$ in humid air and reached the maximum $0.78 \times 10^6 \text{ nm}^3$ in DI water. Comparatively, the removal volume of Ge by SiO₂/Ge pair remained at $\sim 0.16 \times 10^6 \text{ nm}^3$ in different humidity conditions and suddenly increased to $\sim 0.5 \times 10^6 \text{ nm}^3$ in DI water. This indicates that the material removal on the Ge surface by SiO₂ tip shows a highly water-dependent mechanochemical behavior. Correspondingly, there are also clear distinctions in the removal volume on monocrystalline Ge by different tips, while the sliding by Al₂O₃ tip leads to the maximum removal volume. From a practical perspective, these nanoscale

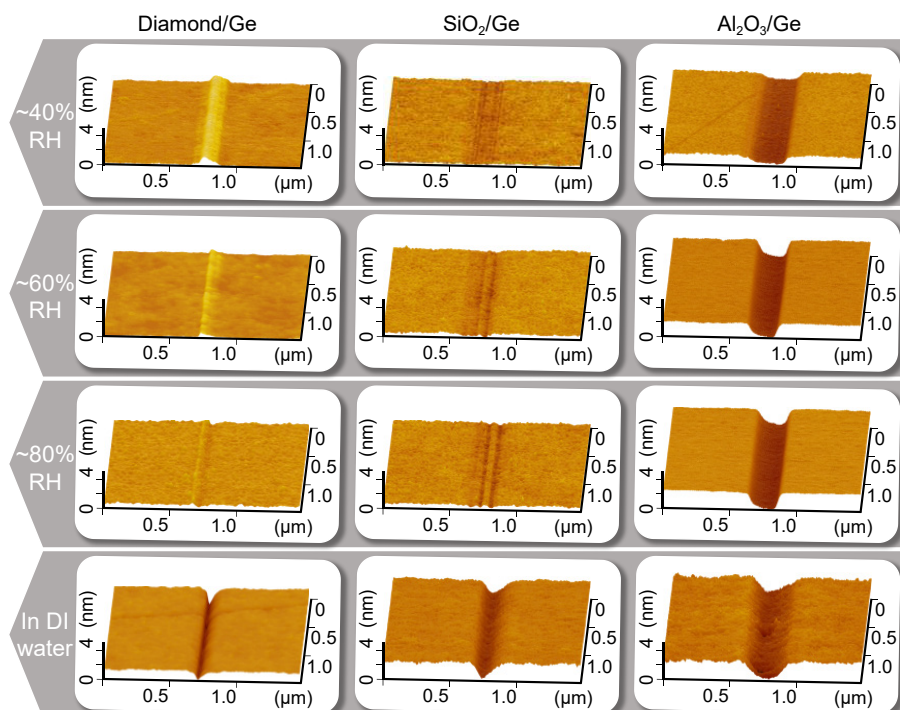


Fig. 2 AFM images of removal tracks on the Ge surface processed by diamond, SiO₂, and Al₂O₃ tips in different ambient environments at $F_n = 4 \mu\text{N}$, $v = 4 \mu\text{m/s}$, and $N = 500$, respectively.

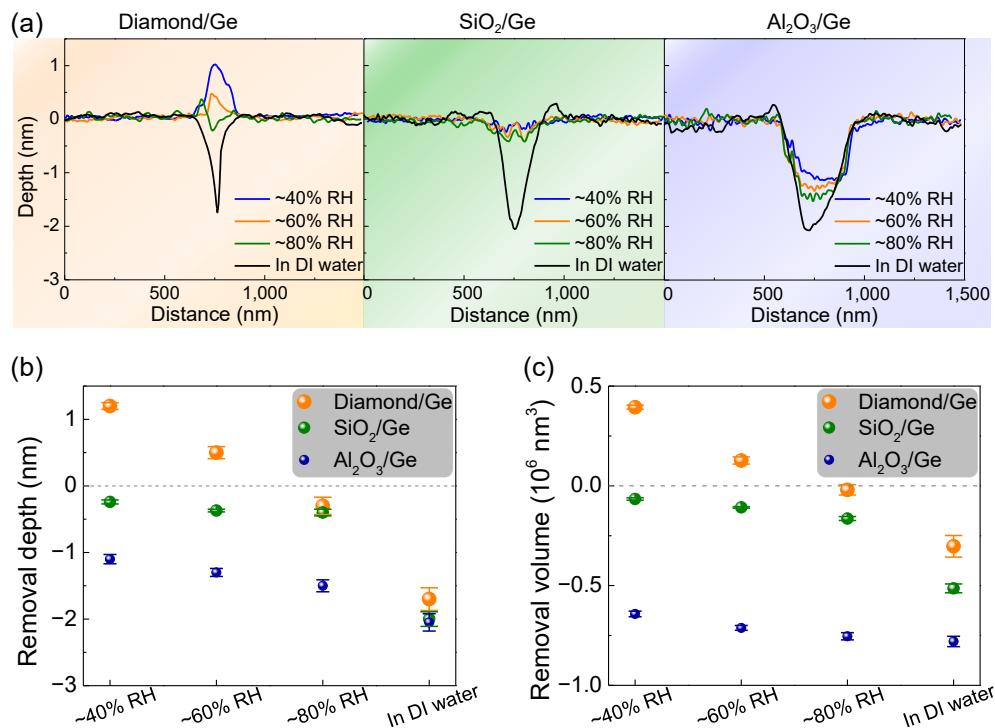


Fig. 3 Comparison of (a) average cross-sectional profiles, (b) removal depth, and (c) removal volume on the Ge surface produced by diamond, SiO₂, and Al₂O₃ tips in different ambient environments at $F_n = 4 \mu\text{N}$, $v = 4 \mu\text{m/s}$, and $N = 500$, respectively.

single-asperity scratch findings establish a fundamental bridge to guide the macroscopic formulation of CMP slurries. The experiments explicitly demonstrate that in an aqueous environment, SiO₂ particles can achieve a remarkably high material removal rate driven entirely by water-assisted tribochemical reactions, effectively circumventing the mechanical processing and subsurface damage typically caused by physical plowing. Consequently, compared to conventional high-hardness abrasives (e.g., diamond or Al₂O₃), SiO₂ particles exhibit significant superiority and emerge as an optimal abrasive candidate for designing advanced CMP slurries tailored for the damage-free planarization of next-generation Ge wafers.

Considering that abrasive particles may participate as reactants in the removal process and the attrition of the tips directly determines their performances, the morphological changes of the tips were also taken into consideration during the tests. AFM topographic images of the three tips before and after the tests are shown in Fig. 4, and they were obtained by a diffraction grating with arrayed (spacing $\sim 3 \mu\text{m}$) silicon tips of curvature radius $\leq 10 \text{ nm}$. It can be observed from the AFM topographic images that the SiO₂ and Al₂O₃ tips are microspheres with a curvature radius of about $1 \mu\text{m}$. After the tests, the SiO₂ tips present obvious attrition by comparing the corresponding cross-section profile, followed by Al₂O₃ tip. Under similar contact pressure during the nanoprocessing test, the Al₂O₃ tip sliding seems to cause more substrate removal than tip attrition, causing more material removal from the Ge surface. Due to the highest hardness of the diamond, the morphology of the diamond tip is almost unchanged after the tests. This suggests that SiO₂ and Al₂O₃ tips may partially participate in the reaction during the mechanochemical removal process.

3.3 Microstructure defect detection

It was reported that nanoscratches produced by diamond sliding can induce different paths of crystal deformations on Ge substrates, hence, the material removal on Ge surface by diamond

tip sliding was often accompanied by defects [33]. The detection of the phase transformation in the processed zone by Raman spectroscopy can be applied to understand whether the defect exists on the Ge surface during the material removal process. As shown in Fig. 5, the spectral signal curves gathered in the processed area were produced by SiO₂, Al₂O₃, and diamond tips. The orange spectral curve represents the scratch generated by diamond tip. It can be observed that a broad spectral band component below 290 cm^{-1} as well as a sharp and narrow peak at $\sim 300 \text{ cm}^{-1}$ were present in the spectral curve, which indicates that significant amounts of amorphous Ge appear in addition to the crystalline structure [33]. Nevertheless, the spectral curves produced by SiO₂ and Al₂O₃ tips are consistent with the spectral curve of pristine Ge. This result illustrated that SiO₂/Ge and Al₂O₃/Ge pairs might not be accompanied by the defects on the Ge surface during the material removal process.

To further understand the nanostructure of the scratched zone and reveal the interfacial material removal behavior on the Ge surface, the cross-sections of the scratches on the Ge surface were characterized by HRTEM. Figure 6 shows the HRTEM images of the scratched areas produced by diamond, SiO₂, and Al₂O₃ tips in humid air, respectively. A HRTEM image of the hillock produced by diamond tip under $4 \mu\text{N}$ is displayed in Fig. 6(a), and the image Fig. 6(a₁) shows the magnified view of the misorientation at the bottom of the hillock. Dark zones reflecting lattice distortions can be observed in the orange dotted-line square, and the inserted picture provides the corresponding inverse fast Fourier transformation (IFFT) image for presenting dislocations [34]. Figures 6(b) and 6(c) show the removed region generated by SiO₂ and Al₂O₃ tips under $4 \mu\text{N}$, and no distinct disordered Ge lattice was observed anywhere within the scratched regions. The result illustrates that the surface crystal lattice and atomic arrangement would not be distorted by SiO₂ and Al₂O₃ tips. This demonstrates that no defects (e.g., crystal lattice distortion and dislocation) were generated on the Ge surface during the sliding process by SiO₂ and Al₂O₃ tips compared to the sliding by diamond tip, indicating

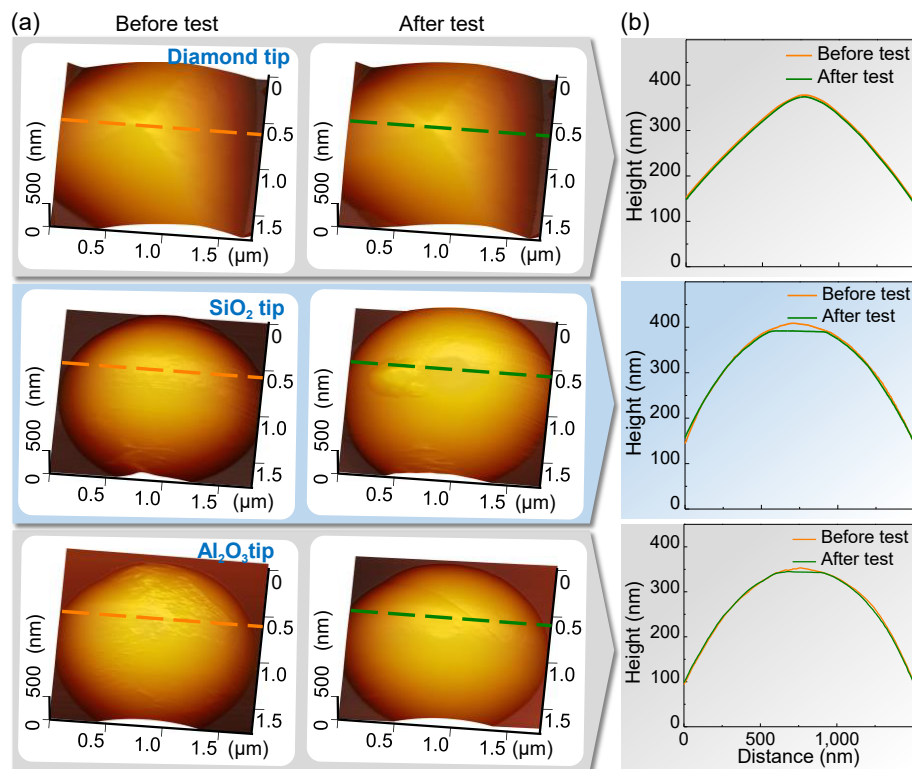


Fig. 4 Topography changes of diamond, SiO₂, and Al₂O₃ tips before and after the tests.

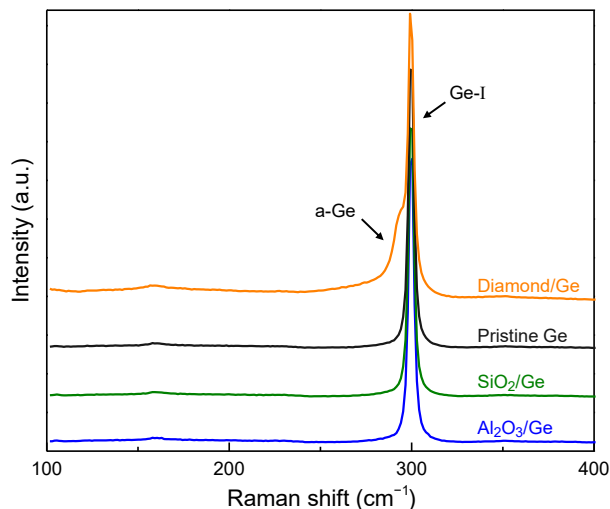


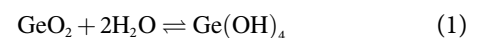
Fig. 5 Comparison of micro-Raman spectra obtained from different 20 μm × 20 μm-square scratches (prepared under 40 μN by diamond tip, and 4 μN by diamond, SiO₂, and Al₂O₃ tips, respectively), and pristine Ge surface.

that the tribochemical interaction between SiO₂/Ge and Al₂O₃/Ge interface dominated the defect-free removal. In summary, the defect-free removal behavior on the Ge surface might involve only the outermost layer of the interfacial contact, and surface atoms were removed under compressive and shear stress, while the deeper atoms remained almost unaffected and the crystal lattice also maintained its integral perfectly.

3.4 Mechanochemical interaction

The lattice structure of monocrystalline Ge is quite similar to that of Si, so the mechanochemical removal mechanism on the Ge surface can refer to that of Si. The removal of Si first involves the

polarization of surface Si-Si bonds by hydroxyl ions, followed by the attack of water molecules and dissolved oxygen [35]. Based on this, the mechanochemical reaction process at Ge/SiO₂ interface can be addressed below as an example. Ge-Ge backbone bonds were weakened by oxidation due to the presence of hydroxyl ions and radicals under neutral conditions, resulting in enhanced hydrolysis and subsequent removal [36]. Both SiO₂ tip and Ge surface could be chemically modified by hydroxylation with the participation of the adsorbed water film, resulting in hydroxy termination on the contacting surfaces. That is, Ge-OH groups are formed on the Ge surface, while Si-OH dangling groups are formed on the SiO₂ tip. The dehydration and condensation reactions occur between Ge-OH and Si-OH groups at the sliding Ge/SiO₂ interface, forming “Ge-O-Si” bonding bridges. The bond strengths of Ge-Ge and Ge-O bonds are 37.6 and 86 kcal/mol, respectively [36], and the weaker Ge-Ge bond can be more easily broken during the relative sliding of SiO₂ tip and Ge surface. More fundamentally, this bond dissociation is a dynamic, stress-driven mechanochemical activation process [37]. The localized shear stress generated at the single-asperity contact significantly lowers the chemical activation energy required for breaking the Ge-Ge backbone bonds. Therefore, the continuous breaking of Ge-Ge bond leads to the gradual removal of Ge from the substrate during the sliding process. The removed Ge from the surface will be rapidly oxidized to GeO₂, and further aqueous phase equilibrium reactions will occur (Eq. (1)) [18].



Particularly in a fully submerged DI water environment, the bulk water acts as both an abundant reactant and an efficient mass transport medium. Because GeO₂ exhibits exceptional water solubility, the surrounding water rapidly dissolves these localized oxidized products, thereby preventing surface passivation and continuously exposing fresh Ge lattices for ongoing

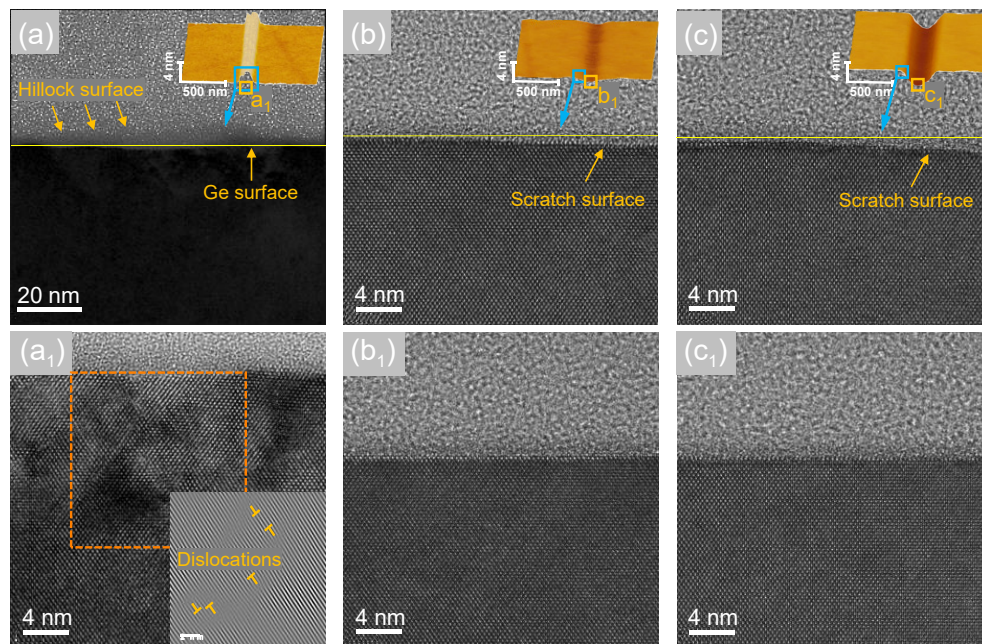


Fig. 6 HRTEM image of the cross-section microstructure of scratches on the Ge surface created by (a) diamond, (b) SiO₂, and (c) Al₂O₃ tips, respectively. (a₁–c₁) display the images of the bottom of the scratch corresponding to the marked area in the inserted AFM images in (a–c), respectively.

mechanochemical cycles. As a result, the water can facilitate material removal from the Ge surface. Correspondingly, the material on SiO₂ tip may also show light attrition due to the weakening effect of hydroxide ions (see Fig. 4). Similar behaviors may occur when other covalent and highly polar materials, such as sapphire (Al₂O₃) [38], are brought into tribological contact with Ge surface.

4 Conclusions

In this study, nanoprocessing tests were carried out with atomic force microscope (AFM) tips of different materials on the monocrystalline Ge surfaces, and the main points are summarized as follows:

(1) Under purely elastic contact conditions, the mechanochemical removal was achieved by SiO₂ and Al₂O₃ tips on monocrystalline Ge surfaces in humid air, respectively. In contrast, the friction-induced hillocks can be generated during diamond tip sliding at low humidity.

(2) Ge exhibited distinct material removal when scratched by the three tips (SiO₂, Al₂O₃, and diamond tips) at different humidities and in deionized (DI) water, while the Al₂O₃ tip caused the largest amount of material removal.

(3) Combined with micro-Raman spectra and HRTEM detection, the defect-free removal on the Ge surface by SiO₂ and Al₂O₃ tips was confirmed to be dominated by mechanochemical interactions. For comparison, mechanical actions play a key role in material removal on the Ge surface when diamond tip is employed.

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Declaration of competing interest

The authors have no competing interests to declare that are relevant to the content of this article. The author Linmao Qian is an Editorial Board member of this journal.

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